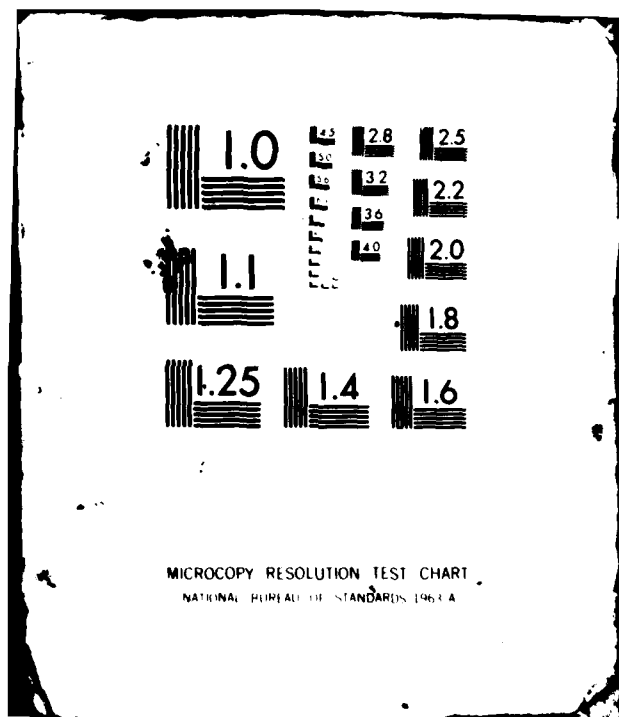


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QUALIFICATION OF A NEW MAPO SOURCE AND ERL-510 CURING AGENT FOR--ETC(U)
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UNITED STATES AIR FORCE

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QUALIFICATION OF A NEW MAPO
SOURCE AND ERL-510 CURING AGENT
FOR MINUTEMAN STAGE I UF-212I LINER

PROPELLANT ANALYSIS LABORATORY

MANPA REPORT

NR 464(82)

JANUARY 1982

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MANPA REPORT
NR 464(82)

QUALIFICATION OF A NEW MAPO
SOURCE AND ERL-510 CURING AGENT
FOR MINUTEMAN STAGE I UF-2121 LINER

Author

John A. Thompson
JOHN A. THOMPSON, Chemist
Component & Combustion Test Unit

Engineering & Statistical Review By

John K. Scambia
JOHN K. SCAMBIA, Project Engineer
Service Engineering

Dan L. Petersen
DAN L. PETERSEN, Mathematician
Data Analysis Unit

Recommended Approval By

Leonidas A. Brown
LEONIDAS A. BROWN, Chief
Component & Combustion Test Unit

Approved By

Anthony J. Inverso
ANTHONY J. INVERSO, Chief
Propellant Analysis Laboratory

January 1982

Ind Products & Ldg Gear Division
Directorate of Maintenance
Ogden Air Logistics Center
United States Air Force
Hill Air Force Base, Utah 84056

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ABSTRACT

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Thiokol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required.

Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.

This report includes the test results for the first through the seventh time testing of the control and special specimens at Ogden ALC.

Statistical analysis of the test data showed that the physical properties of the new source compared closely to the old source of UF-2121 liner specimens.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 REV A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

TABLE OF CONTENTS

	<u>Page</u>
Abstract	11
Introduction;	
Purpose	1
Background	1
Table 1, Test Conditions and Methods	3
Statistical Analysis	4
Test Results	6
Conclusions and Recommendations	8
Table 2, Test Data Summary (1975)	9
Table 3, Test Data Summary (1976)	9
Table 4, Test Data Summary (1977)	9
Table 5, Test Data Summary (1978)	10
Table 6, Test Data Summary (1979)	10
Table 7, Test Data Summary (1980)	10
Table 8, Test Data Summary (1981)	11
Table 9, Analysis of Covariance Results	11
Figure 1, Control Disc (Steel/Liner/Steel)	12
Figure 2, Special Disc (Steel/Liner/Steel)	13
Figure 3, Control Cup (Steel/Liner/Steel)	14
Figure 4, Special Cup, (Steel/Liner/Steel)	15
Figure 5, Control Peel (Steel/Liner/Steel)	16
Figure 6, Special Peel (Steel/Liner/Steel)	17
DD-1473	18
Distribution List	20

INTRODUCTION

A. PURPOSE:

Quality assurance testing of specimens prepared from the new source of MAPO to assure that liner material for First Stage Minuteman Motors will perform as predicted.

B. BACKGROUND:

*Tris [1-(2 methyl) aziridinyl] phosphine oxide (MAPO) is used as a curing agent in the Minuteman Stage One UF-2121 liner. MAPO was produced by Immont Chemical (Immont) and shipped to Arsynco Incorporated (Arsynco) for purification and marketing. Immont sold the production rights for MAPO to Arsynco and terminated the production of raw MAPO in 1972.

Since MAPO is a critical ingredient in UF-2121 liner formulation, the source change for the manufacture of MAPO was considered a serious change. Therefore, it was necessary for Thiokol to conduct qualification testing on liner material using MAPO manufactured by Arsynco before it could be considered acceptable for use in Minuteman Stage I UF-2121 liner.

ERLA-500 was the qualified epoxy curing agent used with MAPO in the UF2121 liner. Union Carbide terminated their process for ERLA-500. ERLA-510 used in similar liners (i.e. UF-2137) was substituted for ERLA-500 and qualified with MAPO from the new source.

The test conditions and test methods are shown in Table I.

*TWR-7857 Rev A Report, J. W. Rabern

Qualification testing was performed by Thiokol and reported in TWR-7857 Rev A. In addition, specimens were prepared by Thiokol from new vendor's material and from the old source material for a "follow on" test program. These specimens were then transferred to Ogden ALC for a continuing surveillance test program designed to cover a ten year span. The material from the old source will be used as the control samples.

The ten year sampling plan is shown below. Those specimens identified for the seventh year were tested at this test period. The types of specimens are Disc (steel/liner/steel), Cup (steel/liner/TP-H1011), and Peel (broadcloth/liner/TP-H1011). For the disc specimen, the adhesion between the liner and steel is the critical factor. For the cup specimen, the adhesion between the propellant and the liner is critical. For the peel specimen the propellant to liner peel strength when pulled at 180°F is critical.

TEN YEAR CONTINGENCY AGING SAMPLE CODING

Age (yr)	Storage Temp (°F)	Disc (Sample Nr)		Cup (Sample Nr)		Peel (Sample Nr)	
		Control*	Special**	Control*	Special**	Control*	Special**
1	75	1 - 6	181-186	61-66	241-246	121-126	301-306
2	75	7-12	187-192	67-72	247-252	127-132	307-312
3	75	13-18	193-198	73-78	253-258	133-138	313-318
4	75	19-24	199-204	79-84	259-264	239-144	319-324
5	75	25-30	205-210	85-90	265-270	145-150	325-330
6	75	31-36	211-216	91-96	271-276	151-156	331-336
7	75	37-42	217-222	97-102	277-282	157-162	337-342
8	75	43-48	223-228	103-108	283-288	163-168	343-348
9	75	49-54	229-234	109-114	289-294	169-174	349-354
10	75	55-60	235-240	115-120	295-300	175-180	355-360

* Liner mix A73-11846 - control or old MAPO source material

** Liner mix A73-11810 - Experimental MAPO

TABLE I

Test Conditions and Methods

<u>Group</u>	<u>Test</u>	<u>Condition</u>	<u>Config- uration</u>	<u>G085 Spec Code</u>	<u>Spec Per Cond</u>	<u>Total Number of Spec</u>	<u>Test Method</u>
Bond in Tension Disc	Tensile Adhesion OI#127-3	CHS 0.5 in/ min, Chart 5.0 in/min, 500 lbs full scale load $77^{\circ}\text{F} \pm 2^{\circ}$	Discs	TV	Control 6 Special 6	12	A
Bond in Tension Cup	Tensile Adhesion OI#127-3	CHS 0.5 in/ min, Chart 5.0 in/min, 200 lbs full scale load $77^{\circ}\text{F} \pm 2^{\circ}$	Cup	TC	Control 6 Special 6	12	A
180° Peel Specimens	Tensile Peel OI#127-3	CHS 10 in/min $77^{\circ}\text{F} \pm 2^{\circ}$ Chart 5 in/ min 20 lbs full scale load	Peel	TE	Control 6 Special 6	12	B

TEST CONDITIONS

A. Testing of tensile adhesion specimens was performed using an Instron testing instrument. Properties measured were maximum stress to the nearest pound and failure mode.

Steel disc specimens require a stress of about 240 psi. The recommended initial full scale load is 500 pounds. This instrument setting should be changed to another setting if the first reading goes off scale on the high side. If 500 psi is exceeded, then change the reading to 1000 psi full scale.

Cup adhesion specimens are tested with a stress of 200 lbs per sq inch; the recommended full scale load is 500 lbs.

B. Testing of 180° peel samples was performed using an Instron testing instrument. The physical property of the material to be determined was the average peel strength to the nearest pound per inch.

NOTE: Thiokol's procedure for Testing and Laboratory Mixing of UF-2121 Liner. SLP 400, 28 April 71.

STATISTICAL ANALYSIS

UF-2121 liner material is being tested under a ten-year program to determine whether or not differences exist between liner materials manufactured from two separate sources of curing agent (MAPO). Test specimens were manufactured in two groups; control, using original source curing agent, and special, using new source curing agent. The test specimens for these two groups are of three kinds; disc, cup, and peel. For each specimen type within each test group the sample test size is six. Laboratory testing for seven test periods or seven years has been accomplished. Test data for the years 1975 through 1981 are contained in Tables 2 through 8, and columns are summarized using means and standard deviations.

With seven test periods accomplished, regression plots (Figures 1 thru 6) were made to determine whether slope and elevation differences existed between control and special test data. No differences in either slope or elevation were found. The regression model $Y = a + bX$, using individual data points, was used in the regression analyses. The variance about the least squares trend line is used to compute a tolerance interval such that at the 90% confidence level 90% of the sample distribution falls within this interval. This tolerance interval is extrapolated 24 months past the age point pertaining to the oldest specimens tested. The statistical significance of the slope of the trend line is evaluated for each regression plot. If significant, it is an indication that change over time is occurring.

In determining differences in data pertaining to the two MAPO sources, analysis of covariance was employed to compare control and special data from

the regressions for each of the three types of test specimens. For analysis of covariance results, see table 9. Taken at the five percent significance level, the only difference found was in the disc data in the variance of the data away from the regression line.

TEST RESULTS

The 1981 test data and the means for the respective control and special data are shown in Table 8. In addition, for a convenient comparison, the 1975 through 1980 test data are included in Tables 2 through 7.

The statistical analysis results for the 1981 testing are shown in Table 9 with the regressions shown in Figures 1 through 6.

DISC:

A statistically significant difference is shown for variance of test data in the MAPO source (Table 9) with no significant difference for the slope and elevation of the regression curves (Table 9).

The regression curves show a statistically significant gradual decrease for both the control and special sample data (Figures 1 and 2).

For the year 1981, the mean of the control and special data is 15.49 and 16.07 kg/sq cm respectively.

The minimum specification requirement according to TWR-7857 REV A, is 12.30 kg/sq cm minimum. As seen in Table 8, MANPA's data is well above this minimum.

The failure mode was 100% cohesive in the liner for both the control and special specimens.

CUP:

There is no significant difference in variance, slope or elevation when comparing control and special regression data (Table 9).

The regression curves show a statistically significant gradual decrease in maximum stress as the specimens age (Figures 3 and 4).

According to TWR-7857 REV A Report, the minimum requirement is 4.92 kg/sq cm. The data means are 9.37 kg/sq cm for the control and 9.21 kg/sq cm for the special specimens.

For the control specimens, the failure mode for 4 specimens was 100% cohesive in the propellant, one specimen 66% cohesive in the propellant and 34% adhesive liner to propellant with the remaining specimen 80% cohesive in the propellant and 20% adhesive liner to propellant.

For the special specimens, three had 100% cohesive failure in the propellant, two specimens had 70% cohesive failure in the propellant and 30% adhesive liner to propellant failure and the remaining specimen failure mode was 45% cohesive in the propellant and 55% adhesive liner to propellant.

PEEL:

No significant difference is shown for the variance, slope or elevation when comparing control with special regression data (Table 9).

The regression curves (Figures 5 & 6) do not show a statistically significant difference in peel strength with respect to the age of the specimens.

Thiokol reported (TWR-7857 REV A) 0.679 and 0.732 *kg/L cm respectively for the control and special mean data at age six months. This compares with 1981 data of 0.81 and 0.85 kg/l cm respectively for control and special mean data (Table 8).

The mode of failure was 100% liner to propellant bond.

* Kilograms per linear centimeter

CONCLUSIONS

Based on this analysis, the only statistically significant difference between the control and special specimens is the variance for disc specimens.

The disc and cup regressions show a gradual statistically significant decrease with the peel specimen data showing no significant changes.

The strength of the specimens is well above the required minimum for disc and cup, and above that reported in Thiokol's testing for peel.

From the analysis of the data, the new source of raw material performs as well as the old source; and therefore is expected to perform satisfactorily.

RECOMMENDATIONS

It is recommended that the testing plan be continued to assure long range capability of the liner produced from the new source of material.

TABLE 2. TEST DATA SUMMARY FOR JULY 1975

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/L-cm	Nr	Kg/L-cm
1	17.085	181	17.225	61		241	11.601	121	0.6786	301	0.6786
2	17.507	182	17.858	62	11.812	242	11.601	122	0.6786	302	0.6965
3	17.225	183	17.015	63	11.741	243	11.531	123	0.7143	303	0.6965
4	17.929	184	16.944	64	12.163	244	11.671	124	0.7500	304	0.6965
5	17.366	185	17.436	65	12.234	245	11.390	125	0.7679	305	0.7143
6	17.296	186	19.054	66	11.882	246	11.390	126	0.7858	306	0.6965
\bar{Y}	17.401		17.589		11.966		11.531		0.7292		0.6965
S	0.2943		0.7899		0.2191		0.1176		0.0458		0.0113

TABLE 3. TEST DATA SUMMARY FOR MAY 1976

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/L-cm	Nr	Kg/L-cm
7	15.116	187	14.483	67		247	13.288	127	0.9643	307	1.0536
8	16.311	188	14.905	68	13.710	248	13.710	128	0.9286	308	1.0358
9	15.397	189	14.483	69	13.640	249	13.640	129	0.9286	309	1.0179
10	15.960	190	14.765	70	13.007	250	13.077	130	1.0179	310	1.0358
11	15.819	191	15.468	71	13.148	251	13.359	131	1.1072	311	1.0536
12	14.554	192	14.765	72	13.499	252	13.499	132	1.0001	312	1.0358
\bar{Y}	15.526		14.812		13.401		13.429		0.9911		1.0388
S	0.6356		0.3633		0.3088		0.2354		0.0675		0.0134

TABLE 4. TEST DATA SUMMARY FOR APRIL 1977

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/L-cm	Nr	Kg/L-cm
13	17.155	193	17.436	73	9.281	253	9.140	133	0.7322	313	0.7858
14	16.522	194	16.874	74	9.281	254	9.070	134	0.7143	314	0.7858
15	16.874	195	16.944	75	8.999	255	8.999	135	0.6786	315	0.7858
16	17.015	196	17.366	76	9.281	256	9.140	136	0.7500	316	0.7143
17	16.874	197	17.015	77	9.492	257	8.367	137	0.7500	317	0.7500
18	16.874	198	17.015	78	9.281	258	8.789	138	0.6429	318	0.7143
\bar{Y}	16.886		17.108		9.269		8.918		0.7113		0.7560
S	0.2107		0.2337		0.1570		0.2994		0.0429		0.0352

NOTE: Kg/L-cm = Kilograms per linear centimeter. Average peel is given for each peel parameter.

TABLE 5. TEST DATA SUMMARY FOR JUNE 1978

DISC		CUP		PEEL	
Control	Special	Control	Special	Control	Special
Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/L-cm	Nr Kg/L-cm
19 14.906	199 14.554	79 10.898	259 10.406	139 0.9109	319 1.0180
20 14.624	200 14.695	80 10.968	260 10.476	140 0.9823	320 1.0359
21 14.695	201 14.343	81 10.617	261 10.616	141 0.9466	321 1.0180
22 14.906	202 14.343	82 10.125	262 10.125	142 1.0002	322 1.0359
23 14.343	203 14.624	83 10.406	263 10.687	143 1.0716	324 1.0537
24 15.187	204 14.414	84 10.476	264 10.125	144 0.9466	326 0.9287
\bar{Y} 14.777	14.496	10.582	10.406	0.9764	1.0150
S 0.2898	0.1503	0.3166	0.2391	0.0561	0.0444

TABLE 6. TEST DATA SUMMARY FOR JUNE 1979

DISC		CUP		PEEL	
Control	Special	Control	Special	Control	Special
Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/L-cm	Nr Kg/L-cm
25 17.015	205 17.436	85 9.632	265 9.562	145 0.8572	325 0.7679
26 16.874	206 16.874	86 9.703	266 9.140	146 0.8393	326 0.7679
27 16.874	207 16.593	87 9.773	267 9.562	147 0.8572	327 0.9643
28 16.944	208 16.522	88 9.632	268 9.281	148 0.8572	328 0.7143
29 16.804	209 16.382	89 9.632	269 9.421	149 0.7679	329 0.7143
30 16.171	210 17.366	90 9.492	270 9.421	150 0.8036	330 0.7500
\bar{Y} 16.780	16.862	9.644	9.398	0.8304	0.7798
S 0.3070	0.4477	0.0934	0.1644	0.0370	0.0936

TABLE 7. TEST DATA SUMMARY FOR SEPTEMBER 1980

DISC		CUP		PEEL	
Control	Special	Control	Special	Control	Special
Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/L-cm	Nr Kg/L-cm
31 15.116	211 14.764	91 9.281	271 11.109	151 0.9465	331 1.1965
32 14.905	212 14.413	92 10.054	272 10.265	152 1.0179	332 1.1786
33 14.624	213 14.202	93 10.616	273 10.898	153 1.0715	333 1.1072
34 14.905	214 14.272	94	274 10.687	154 1.1072	334 1.0536
35 15.678	215 13.870	95 9.894	275 9.491	155 1.0179	335 1.0358
36 15.116	216 14.272	96 10.054	276	156 1.0715	336 1.0358
\bar{Y} 15.057	14.284	9.998	10.490	1.0388	1.0846
S 0.3539	0.3188	0.4750	0.6397	0.0569	0.0613

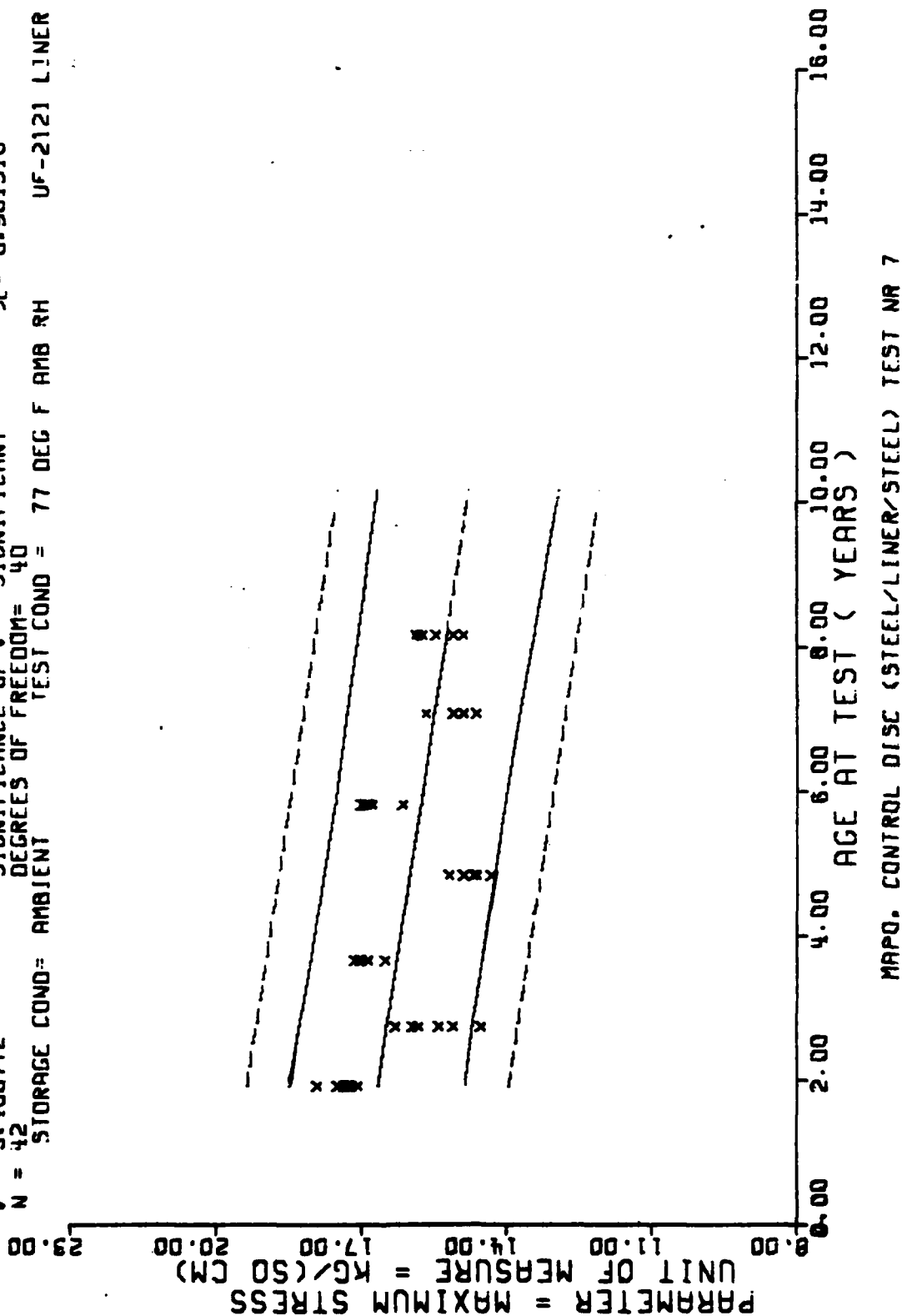
TABLE 8. TEST DATA SUMMARY FOR OCTOBER 1981

DISC			CUP			PEEL		
Control		Special	Control		Special	Control		Special
Nr	Kg/cm ²	Nr Kg/cm ²	Nr	Kg/cm ²	Nr Kg/cm ²	Nr	Kg/L-cm	Nr Kg/L-cm
37	14.905	217 16.874	97	9.421	277 8.999	157		337 0.8036
38	15.819	218 16.944	98	9.210	278 9.070	158	0.7679	338 0.9018
39	15.116	219 15.187	99	9.281	279 9.351	159	0.7858	339 0.8126
40	15.749	220 16.101	100	9.351	280 8.437	160	0.8393	340 0.8304
41	15.890	221 15.679	101	9.632	281 9.562	161	0.8483	341 0.8572
42	15.468	222 15.608	102	9.351	282 9.843	162	0.8215	342 0.8929
\bar{Y}	15.491	16.066		9.374	9.210		0.8126	0.8498
S	0.4044	0.7151		0.1452	0.4911		0.0346	0.0413

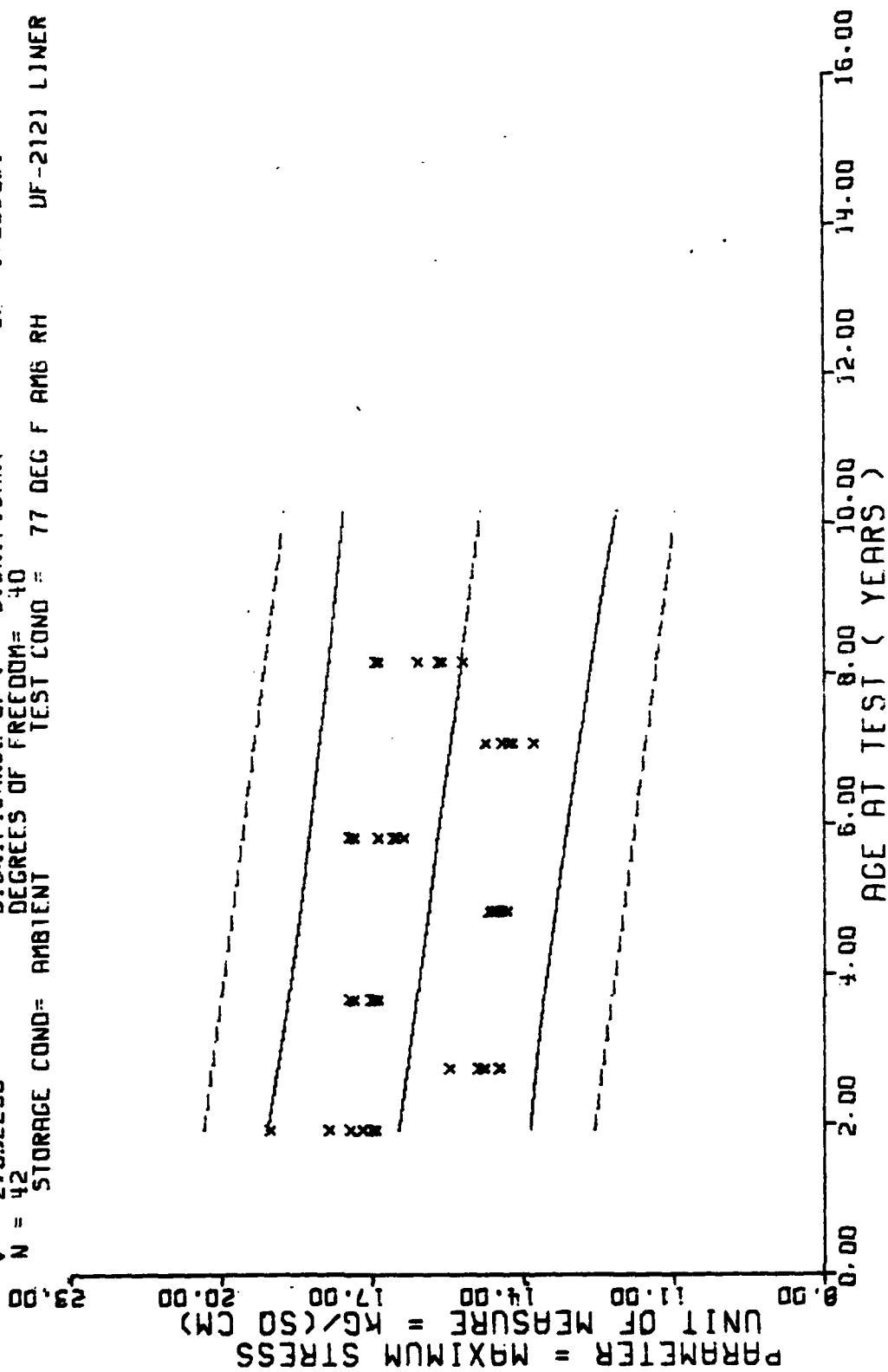
TABLE 9. ANALYSIS OF COVARIANCE RESULTS
COMPARING CONTROL AND SPECIAL REGRESSION DATA

Type of Data Compared	Parameter Compared	F-value	Degrees of Freedom	Significance
DISC	Variance	2.06	40, 40	Significant
	Slope	0.07	1, 80	Not Significant
	Elevation	0.19	1, 81	Not Significant
CUP	Variance	1.38	39, 37	Not Significant
	Slope	0.08	1, 76	Not Significant
	Elevation	0.11	1, 77	Not Significant
PEEL	Variance	1.38	40, 39	Not Significant
	Slope	0.03	1, 79	Not Significant
	Elevation	0.35	1, 80	Not Significant

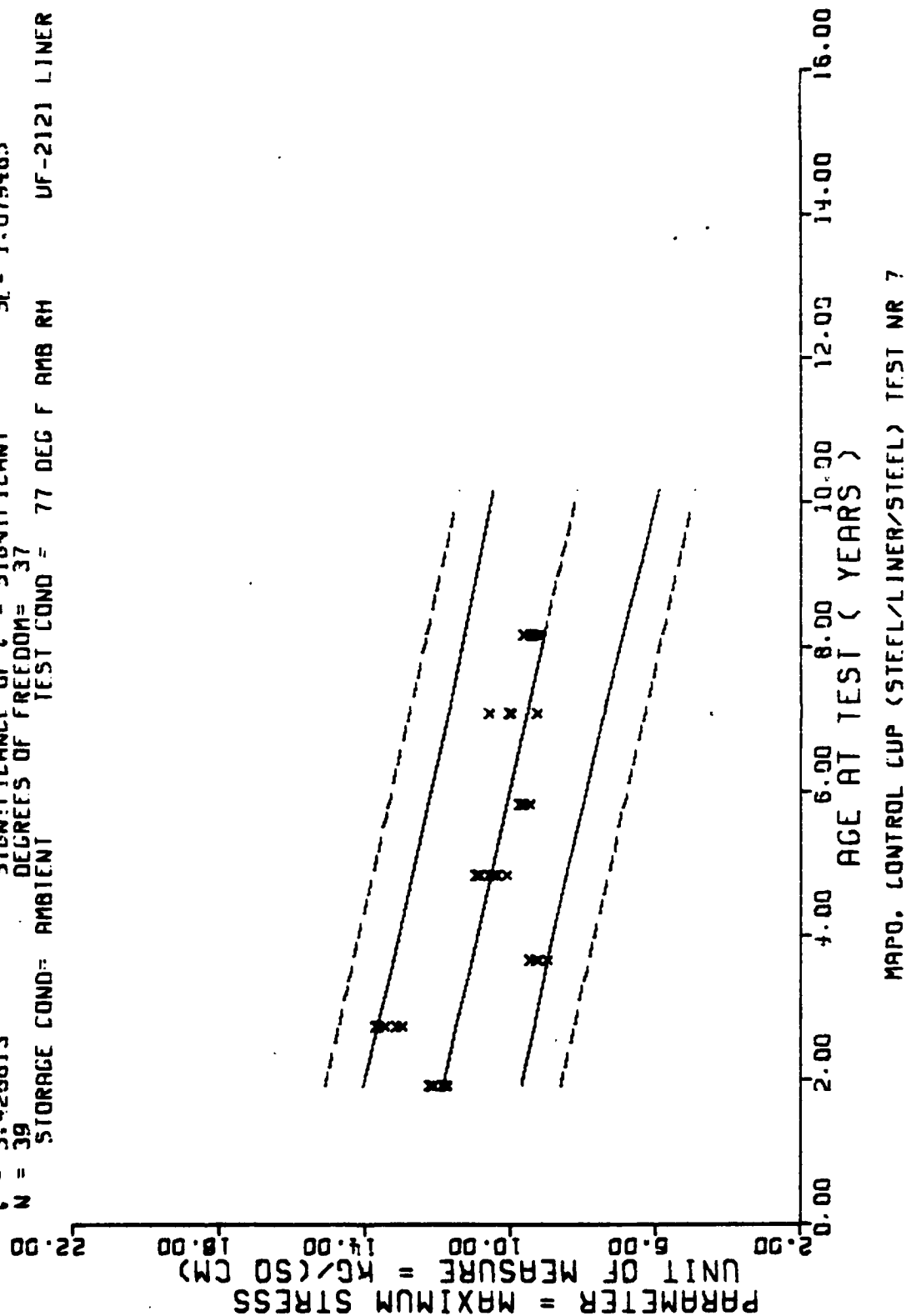
$F = 12.115779$
 $R = -0.482160$
 $t = 3.480772$
 $N = 42$
 $Y = (17.107765) + (-0.019069) \cdot X$
 SIGNIFICANCE OF F = SIGNIFICANT
 SIGNIFICANCE OF R = SIGNIFICANT
 SIGNIFICANCE OF t = SIGNIFICANT
 DEGREES OF FREEDOM = 40
 STORAGE COND = AMBIENT
 TEST COND = 77 DEG F AMB RH UF-2121 LINER



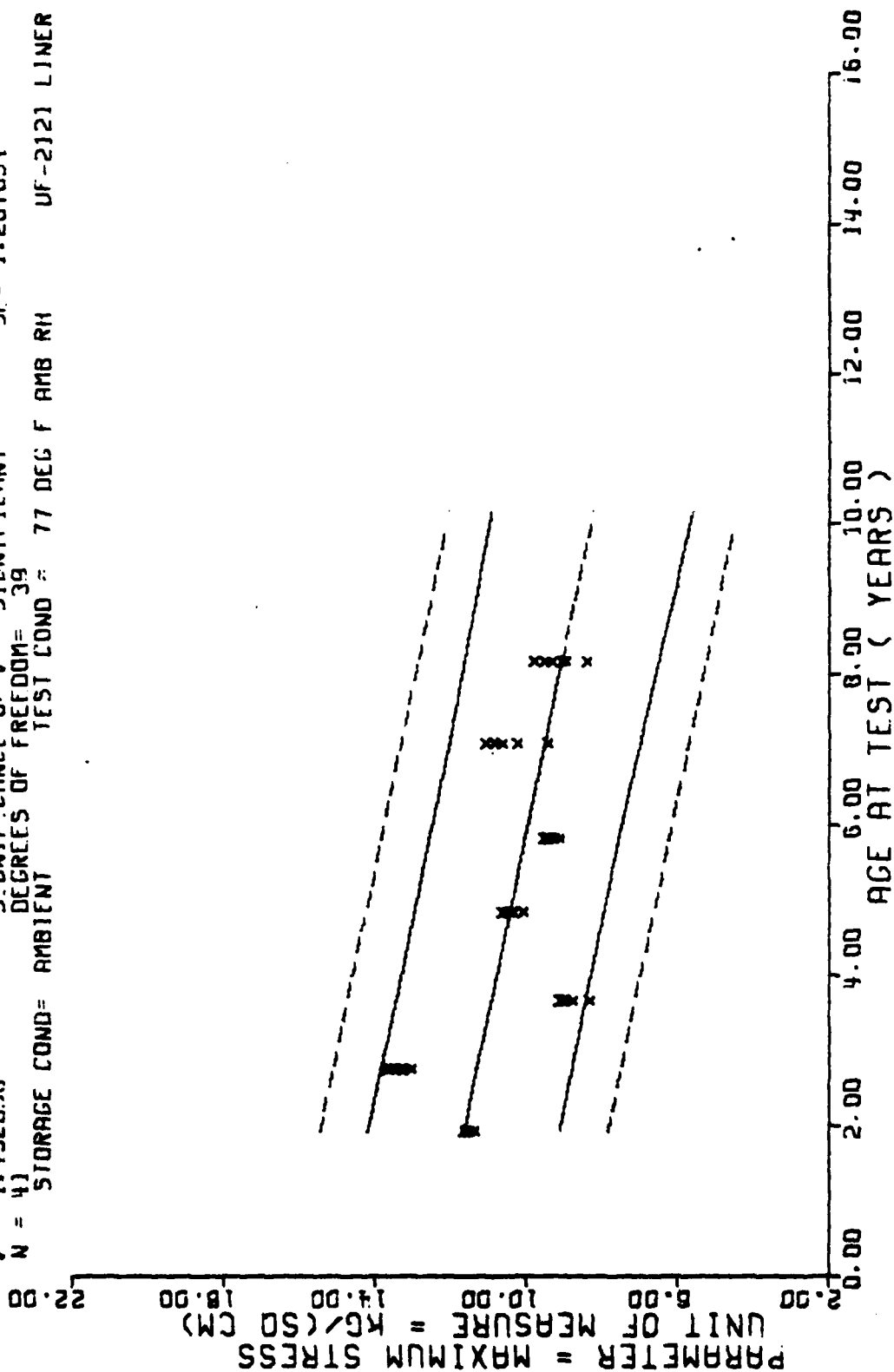
$F = 4.377452$
 $R = -0.314072$
 $t = 2.092236$
 $N = 42$
 STORAGE COND= AMBIENT
 TEST COND = 77 DEG F AMB RH UF-2121 LINER
 $Y = (16.854492) + (-0.016466) * X$
 SIGNIFICANCE OF F = SIGNIFICANT
 SIGNIFICANCE OF R = SIGNIFICANT
 SIGNIFICANCE OF t = SIGNIFICANT
 DEGREES OF FREEDOM = 40
 $Q_1 = 1.347378$
 $S_R = 0.007870$
 $S_E = 1.295091$



$Y = (12.748817) + (-0.037500) \cdot X$
 F = 29.469841
 R = -0.065850
 t = 5.428613
 N = 39
 STORAGE COND= AMBIENT
 TEST COND = 77 DEG F AMB RH
 UF-2121 LINER
 Q_r = 1.427701
 S_e = 0.006908
 SE = 1.079485
 SIGNIFICANCE OF F = SIGNIFICANT
 SIGNIFICANCE OF R = SIGNIFICANT
 SIGNIFICANCE OF t = SIGNIFICANT
 DEGREES OF FREEDOM = 37

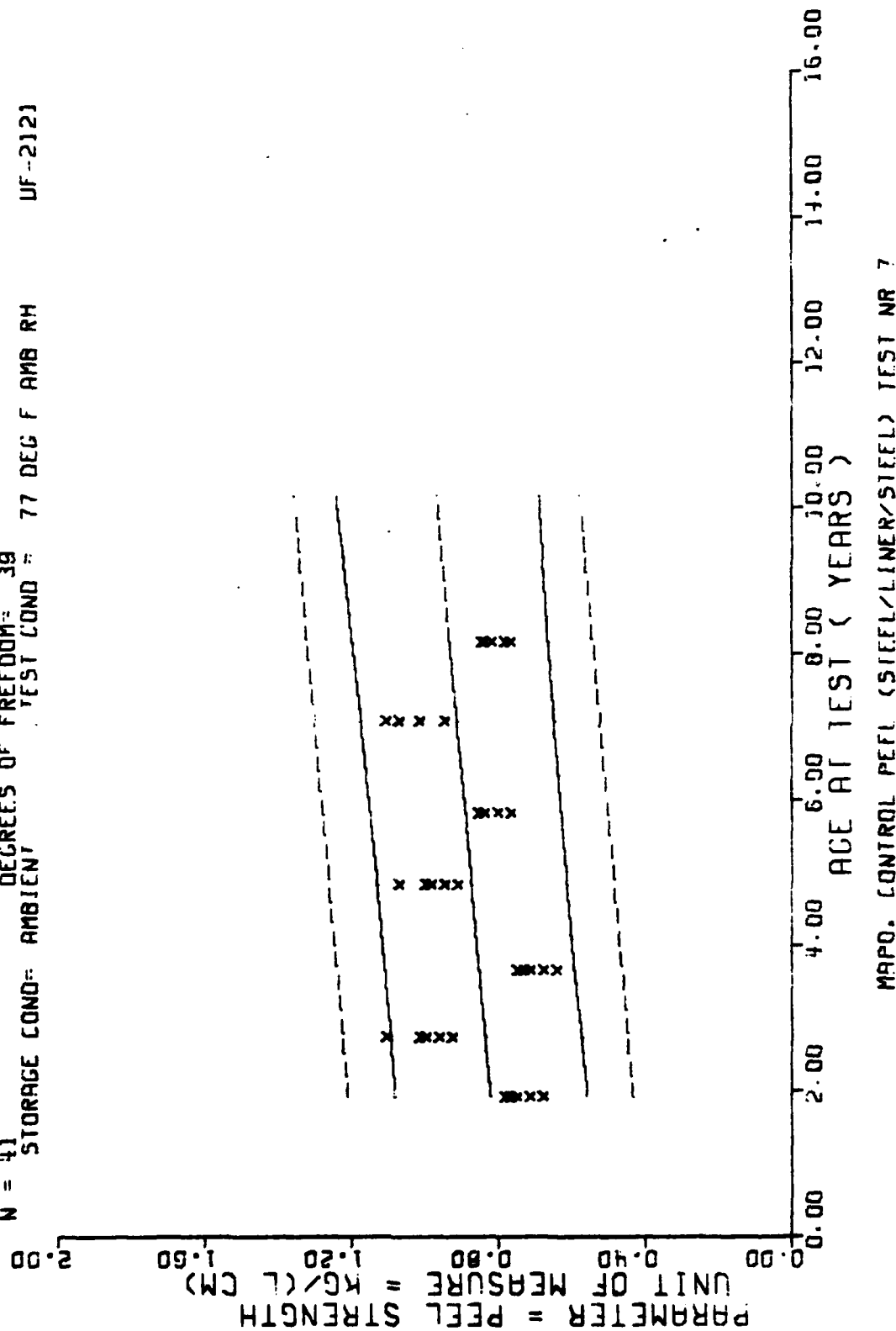


$F = 19.643127$
 $R = -0.578758$
 $t = 4.432056$
 $N = 41$
 STORAGE COND= AMBIENT
 $Y = (12.491516) + (-0.034598) \times X$
 SIGNIFICANCE OF F = SIGNIFICANT
 SIGNIFICANCE OF R = SIGNIFICANT
 SIGNIFICANCE OF t = SIGNIFICANT
 DEGREES OF FREEDOM= 39
 TEST COND = 77 DEG F AMB RH UF-2121 LINER



MAPD, SPECIAL CUP (STEEL/LINER/STEEL) TEST NR 7

$Y = (0.785837) + (0.001481) \times X$
 F = 3.342232
 R = 0.280951
 t = 1.828177
 N = 41
 STORAGE COND= AMBIENT
 DEGREES OF FREEDOM= 38
 TEST COND = 77 DEG F AMB RH UF-2121
 SIGNIFICANCE OF F = NOT SIGNIFICANT
 SIGNIFICANCE OF R = NOT SIGNIFICANT
 SIGNIFICANCE OF t = NOT SIGNIFICANT
 C_V = 0.133102
 S_B = 0.000810
 S_E = 0.129368



F = 3.411114
 R = 0.280316
 t = 1.846920
 N = 42
 STORAGE COND= AMBIENT
 Y = (0.790760) + (0.001708) * X
 SIGNIFICANCE OF F = NOT SIGNIFICANT Qv = 0.156571
 SIGNIFICANCE OF R = NOT SIGNIFICANT Ss = 0.000925
 SIGNIFICANCE OF t = NOT SIGNIFICANT Sx = 0.152161
 DEGREES OF FREEDOM= 40
 TEST COND = 77 DEG F AMS RH UF-2121

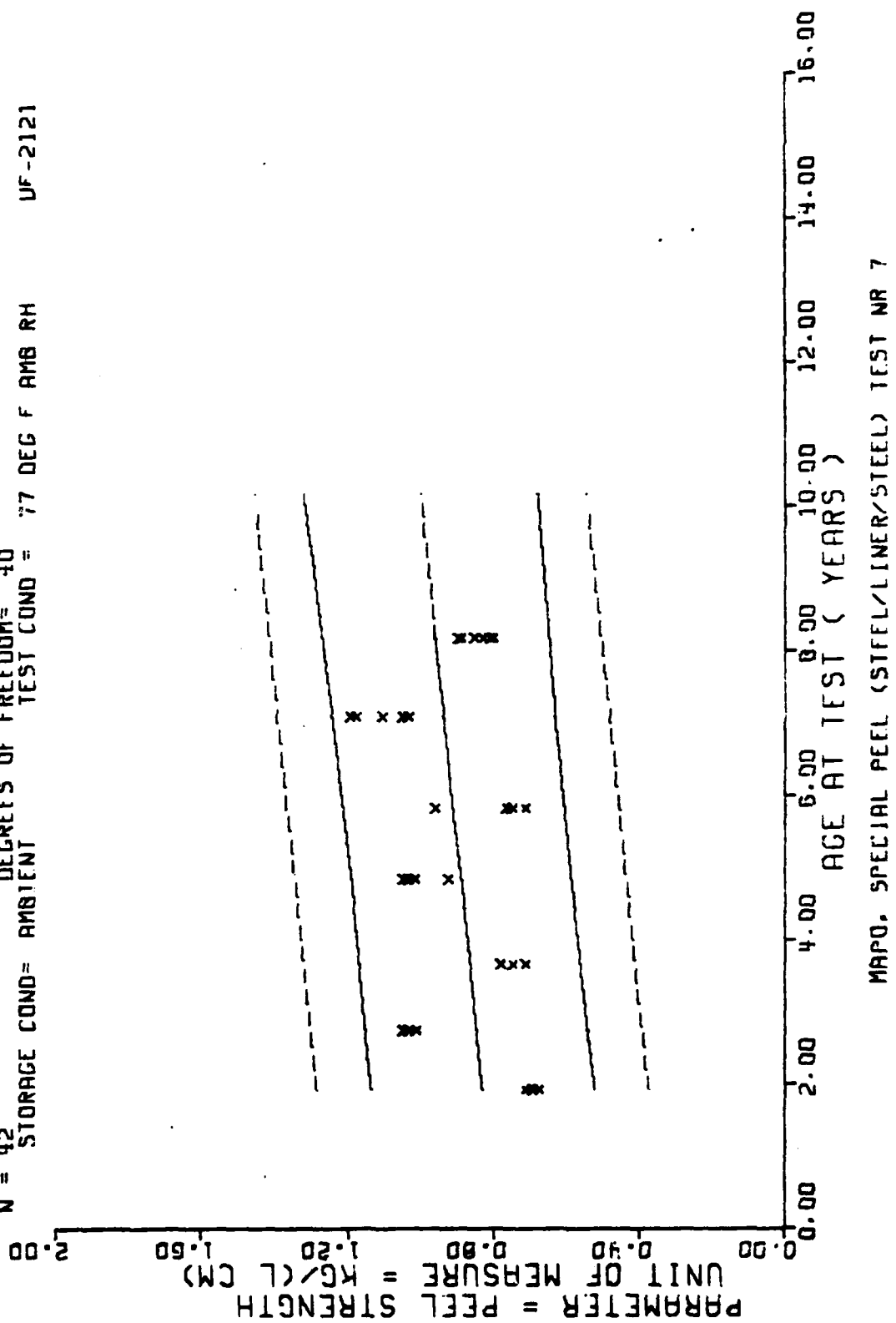


Figure 6

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MANPA REPORT NR 464(82)	2. GOVT ACCESSION NO. AD-A444	3. RECIPIENT'S CATALOG NUMBER 336
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19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Minuteman MAPO UF-2121 liner		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Thiokol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required. Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.		

This report includes the test results for the first through the seventh time testing of the control and special specimens at Ogden ALC.

Statistical analysis of the test data showed that the physical properties of the new source compared closely to the old source of UF-2121 liner specimens.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 REV A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

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AFPRO, Thiokol Chemical Corporation	2
Wasatch Division	
P.O. Box 524	
Brigham City, UT 84302	
(Cy to Larry Hales)	
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